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DEPARTMENT OF CONSUMER AND SENSORY SCIENCES

CONSUMER ODOUR THRESHOLD OF METHYL TERTIARY BUTYL ETHER (MTBE) IN WATER

Work commissioned by: MH3 Corporation

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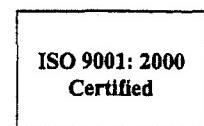
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BACKGROUND

CCFRA was requested to conduct a threshold odour test using consumers, replicating a protocol used by A.J. Stocking *et al.* in a study entitled "Implications of an MTBE odour study for setting drinking water standards", reported in the Journal AWWA, March 2001.

Dr Mel Suffet, MH3 Corporation, observed the work carried out at CCFRA laboratories during 13th-14th August 2003.

OBJECTIVES

To measure odour detection threshold of MTBE in water using 50 untrained consumers.

MATERIALS AND METHODS

2 x 500ml bottles of MTBE supplied by Lyondell Chemicals, Maidenhead, UK.

Still 'Highland Spring' bottled water (2 litre plastic bottles), purchased locally.

Preparation of Test Solutions

All solutions were prepared fresh on each day of testing. The concentrations of MTBE in water used in this study were: $2\mu\text{g.l}^{-1}$, $3.5\mu\text{g.l}^{-1}$, $6\mu\text{g.l}^{-1}$, $10\mu\text{g.l}^{-1}$, $18\mu\text{g.l}^{-1}$, $30\mu\text{g.l}^{-1}$, $60\mu\text{g.l}^{-1}$ and $100\mu\text{g.l}^{-1}$. Analytical confirmation of MTBE concentration in solutions was carried out (Appendix 1). No anomalies were reported.

Test Methods – Odour Threshold with Consumers

A pre-recruited consumer 'central location test' was conducted in three sessions of up to 20 respondents on Wednesday, 13th August and Thursday, 14th August. Respondents were recruited in Evesham and provided with travel to CCFRA. A total of 55 respondents completed the tests.

The following criteria were used to screen the respondents during recruitment:

- Must be non-smokers
- Must not suffer from asthma or any related illnesses
- Must not be pregnant

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- Must not have participated in any food tasting market research tests in the last six months
- The panel of respondents to cover a spread of ages as evenly as possible between the following age groups: 18-29 years, 30-49 years, and 50-64 years
- Gender: 50:50 split
- Social class: no quota

The test was administered according to the procedure published by Stocking *et al.* Each respondent performed eight triangle tests by odour only. The samples were evaluated using the CCFRA Triangle Test Procedure (TES-S-001). In the triangle test each respondent is presented with a set of three coded samples, two of which are the same and one of which is different. Each triangle test paired a water sample with one of the test concentrations. The sets of samples are presented equally often in each of the six possible orders; this experimental design minimises any possible order and carryover effects. After assessing the three samples in the designated order, each respondent was asked to select the different sample. Correct and incorrect responses were then collated.

All assessments were conducted in environmentally controlled sensory booths. The temperature within the booths ranged between 23°C to 25°C over the assessment sessions. The room was positively pressurised to minimise the entrance of external odours and lit by artificial daylight.

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RESULTS

Individual threshold levels are summarised in Table 1. An 'O' represents an incorrect response, a 'X' represents a correct response.

Table 1: Individual Threshold Results by Consumers

Consumer	Concentration ($\mu\text{g.l}^{-1}$)									Consumer	Concentration ($\mu\text{g.l}^{-1}$)								
	2	3.5	6	10	18	30	60	100			2	3.5	6	10	18	30	60	100	
17	0	0	X	0	0	0	0	0	0	118	0	X	0	0	X	0	0	X	
19	0	X	0	X	X	X	X	X	0	117	0	0	X	0	0	X	0	X	
20	X	0	0	X	X	X	X	X	0	108	X	0	X	0	0	X	0	0	
16	X	0	0	0	0	0	0	0	0	107	0	X	0	0	X	X	0	0	
14	0	X	0	0	0	0	0	0	0	102	0	X	X	0	0	0	0	X	
18	X	0	0	X	0	0	X	0	0	101	0	0	0	0	0	X	X	0	
120	X	0	X	0	0	X	0	0	0	106	X	X	0	0	0	0	X	0	
6	X	0	0	0	X	X	X	X	0	105	X	0	0	0	0	0	X	X	
5	0	0	X	X	X	X	X	X	0	103	0	X	0	0	X	0	0	X	
1	X	X	0	0	0	0	X	0	0	104	X	0	0	0	X	0	X	X	
13	X	0	0	X	X	0	0	X	0	204	0	X	X	0	X	X	X	X	
10	0	X	X	X	X	X	X	X	0	207	X	0	0	0	X	X	0	X	
3	0	0	X	0	X	X	0	0	0	210	0	X	0	X	X	X	0	X	
4	X	0	X	X	X	X	X	X	0	211	0	0	X	0	X	X	X	X	
8	0	0	X	0	X	X	X	X	0	212	0	0	0	0	0	X	X	0	
12	X	X	X	X	X	X	0	X	0	209	X	X	X	X	X	0	X	0	
9	0	X	X	X	X	X	X	X	0	208	X	0	0	0	0	X	0	0	
2	0	X	X	0	X	0	0	X	0	214	0	0	0	X	0	0	X	X	
7	X	0	0	0	X	0	X	0	0	201	0	0	X	X	X	X	X	X	
111	0	X	0	X	0	0	0	0	0	205	0	0	X	0	0	0	0	0	
115	0	0	0	0	X	X	X	X	0	203	X	0	X	0	0	0	X	0	
113	0	0	X	0	X	0	X	0	0	213	0	X	0	X	X	X	X	X	
114	0	0	0	0	0	0	X	0	0	202	X	0	0	0	X	X	0	X	
112	0	0	0	X	0	0	0	0	0	215	0	X	0	X	0	0	0	0	
110	0	X	0	X	X	X	X	0	0	206	0	X	X	X	0	X	X	X	
109	X	0	X	X	X	X	0	X	0	216	0	X	X	X	X	0	X	X	
116	X	X	0	X	0	X	0	X	0	217	X	0	0	X	X	X	0	X	
119	0	0	0	X	0	0	0	0	0										

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For each respondent, the geometric mean of the highlighted thresholds was calculated. This is the geometric mean of the last concentration missed, and the first concentration detected, given that all higher concentrations were successfully detected. For those respondents that did not detect the

highest concentration ($100\mu\text{g.l}^{-1}$), their threshold was assumed to be $132\mu\text{g.l}^{-1}$ (the geometric mean of 100 and $175\mu\text{g.l}^{-1}$, which would have been the next highest concentration).

Individual geometric mean concentrations calculated for each consumer ranged from 2.65 to $132\mu\text{g.l}^{-1}$. The test panel geometric mean threshold was calculated to be $52.7\mu\text{g.l}^{-1}$.

The distribution of odour thresholds for the panel is shown in Figure 1 below. Using this graph, it is estimated that the mean threshold of $52.7\mu\text{g.l}^{-1}$ (see Appendix 2) represents the average threshold of approximately 56% of consumers (assuming a lognormal distribution).

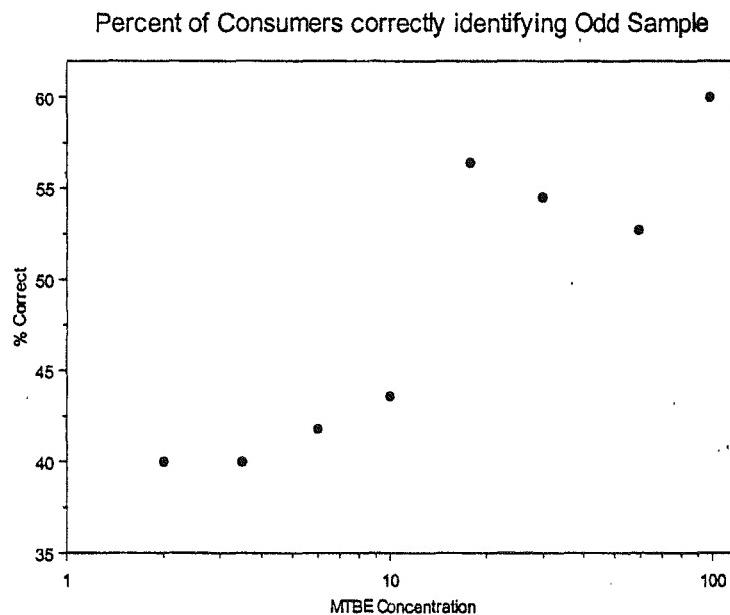


Figure 1: Distribution of Odour Threshold Results

The lower concentrations were not found to be significantly different from water (concentrations 2, 3.5, 6, and $10\mu\text{g.l}^{-1}$), while the higher concentrations ($18, 30, 60$ and $100\mu\text{g.l}^{-1}$) were found to be significantly different. A summary of these results is given in the Table 2.

Table 2: Concentrations Found to be Significantly Different from Water

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Concentration	2	3.5	6	10	18	30	60	100
# Correct	22	22	23	24	31	30	29	33
% Correct	40	40	42	44	56	55	53	60
p-value	0.18176	0.18176	0.11770	0.07175	0.00037	0.00097	0.00234	0.00005
Significance (5%)	NSD	NSD	NSD	NSD	SD	SD	SD	SD

CONCLUSION

For consumers, the odour threshold detection level of $52.7 \mu\text{g.l}^{-1}$ represents the average threshold of approximately 56% of consumers.

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APPENDIX 1

ANALYTICAL CONFIRMATION OF MTBE CONCENTRATION IN SOLUTIONS

All samples were analysed using headspace solid phase microextraction (SPME) and gas chromatography/mass spectrometry in order to determine the concentration of MTBE.

The method used was adapted from a published method (Achten and Puttmann, 2000). Headspace sampling was used instead of immersion in order to prolong the lifetime of the SPME fibre, to ensure that all samples were analysed using the same fibre. It is known that the decision over whether to use headspace or immersion sampling does not have an effect on the result (Pawliszyn, 1999). The use of headspace sampling meant that an increased sampling temperature was beneficial both in terms of sensitivity and repeatability. For that reason, a temperature of 75°C was used. All SPME sampling was carried out by an autosampler, ensuring that all times and temperature were constant throughout the sample set.

METHOD

Extraction of Volatiles by Headspace Solid Phase Micro Extraction (SPME)

Sample (10ml + 2.5g NaCl + 100µl of ethyl acetate solution (1mg.l⁻¹ in water)) was placed into a 20ml vial, and sealed. The headspace of the vial was then sampled for 25 minutes at 75°C (with constant agitation of the vial at a speed of 700rpm) using a carboxen/polydimethylsiloxane coated SPME fibre. The volatiles adsorbed onto the fibre were analysed by thermal desorption at 250°C in the injector port of a GC/MS.

GC/MS Analysis of Volatiles

Analyses were carried out on a Varian 3800 gas chromatograph (GC) and Varian Saturn 2000 ion trap mass spectrometer (MS) via a CTC Combi-Pal autosampler.

GC/MS conditions were as follows:

Column: 25m x 0.25mm fused silica with ZB-624 stationary phase

Helium carrier gas flow rate: 1ml. Min⁻¹

Desorption temperature: 250°C

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Column temperature: 8 min at 50°C; then 25°C. min^{-1} to 250°C, hold for 8 min

MS analysis mode: SCAN 40-100 m/z
 Multiplier offset 200 volts
 Emission current 80 μamps

Quantification of MTBE

MTBE was quantified by comparing the ratio of the integrated chromatographic peak area for MTBE, divided by the peak area for ethyl acetate, with a standard curve generated from the analysis of calibration standards (0.09, 0.9, 4.5, 9, 45 & 90 $\mu\text{g.l}^{-1}$). In order to detect any bias in the preparation of samples and standards, different technicians prepared the samples presented to the consumers and the calibration standards.

RESULTS

Standard Curve

A plot of the ratio of the peak area for MTBE against the peak area for ethyl acetate versus MTBE concentration gave a straight line with an r^2 value close to 1 (Figure A1).

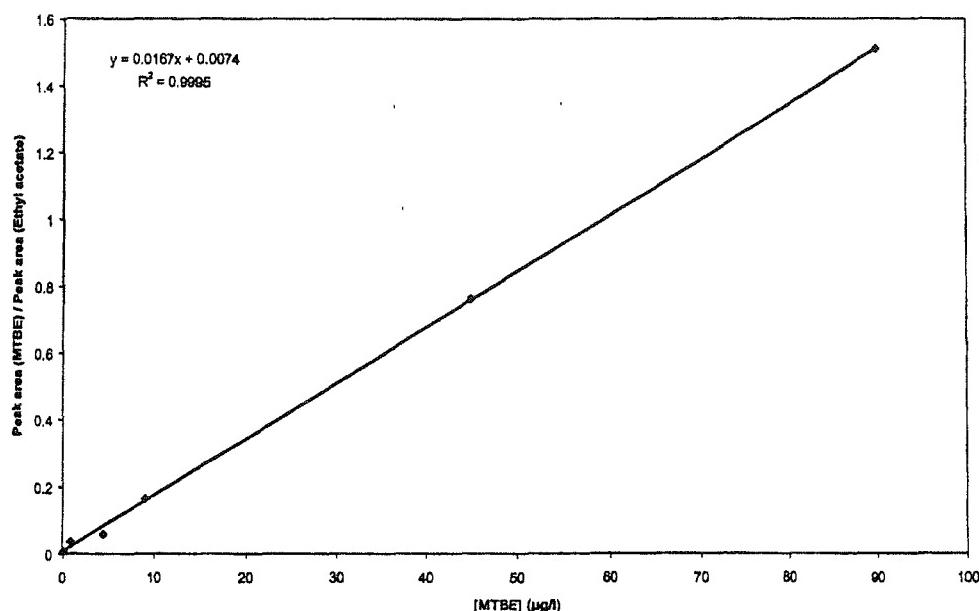


Figure A1: Standard Curve from the Analysis of MTBE Calibration Standards

Results

At low concentrations, it was not possible to determine an accurate concentration for MTBE due to the increased significance of noise and the y-intercept value on the standard curve. Therefore, the limit of quantification was defined as $1 \mu\text{g.l}^{-1}$. This compares favourably with limits quoted for established methods.

Data from the determination of MTBE in samples at or above $1 \mu\text{g.l}^{-1}$ show that the measured concentration of MTBE was close to the nominal concentration in all cases (Table A1).

It must be noted that this type of data is subject to uncertainty from a number of sources including differences between technicians, changes to the sample during storage, sampling errors (i.e. measurement of sample and internal standard volumes) and unavoidable analytical uncertainties. Therefore, the level of agreement between nominal and measured concentrations is very good.

Although it was not possible to accurately quantify MTBE in the samples with concentrations below $1 \mu\text{g.l}^{-1}$, it was appropriate to compare the ratio of MTBE peak area to ethyl acetate peak area across the entire sample set (Figure A2). It was apparent that the instrumental response had a high correlation to nominal concentrations, showing that no anomalies had occurred in the preparation of the samples.

**Table A1: Measured Concentration of with Concentrations
above the Limit of Quantification of $1\mu\text{g.l}^{-1}$**

Date	Nominal Concentration ($\mu\text{g.l}^{-1}$)	Measured Concentration ($\mu\text{g.l}^{-1}$)
12 August	1	-
12 August	10	12
12 August	100	90
13 August*	2	2
13 August*	3.5	3
13 August*	6	6
13 August*	10	10
13 August*	18	16
13 August*	30	29
13 August*	60	46
13 August*	100	79
13 August	1	2
13 August	10	7
13 August	100	83
14 August*	2	2
14 August*	3.5	4
14 August*	6	10
14 August*	10	13
14 August*	18	16
14 August*	30	28
14 August*	60	44
14 August*	100	94
14 August	1	1
14 August	10	15
18 August	1	1
18 August	10	7
18 August	100	106
19 August	1	1
20 August	1	1
20 August	10	7

* Indicates sample presented to consumer panel

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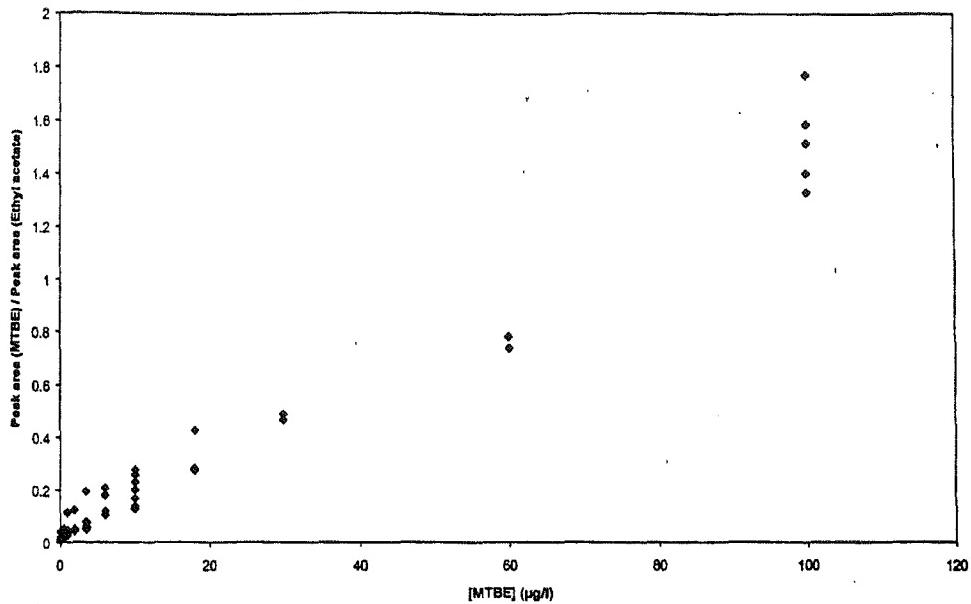


Figure A2: Plot of Instrumental Response against MTBE Concentration

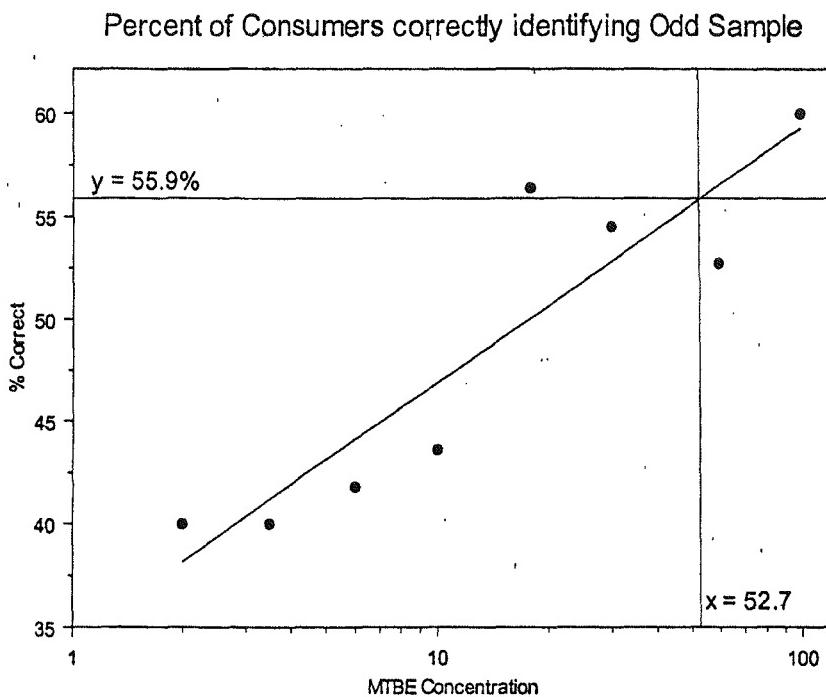
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- Pawliszyn, J. (1999) Quantitative Aspects of SPME. In: *Applications of Solid Phase Microextraction*(Ed, Pawliszyn, J.) Royal Society of Chemistry, Cambridge, pp. 3-21.

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APPENDIX 2

The following was used to determine the percentage of consumers with average concentration of $52.7 \mu\text{g.l}^{-1}$. It represents the data as shown in Figure 1, with a lognormal curve fit to the data.



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